



Synthesis and Characterization of Pure and Capped Zinc Oxide Nanoparticles using *Pesidium Gujava* Leaf Extract

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ABSTRACT

Nanotechnology is a promising concept in the field of science and technology. In this present work, the ZnO nanoparticles were synthesized with and without *Pesidium guajava* leaf extract by using the Microwave irradiation method. The formation of zinc oxide nano particles has been characterized by various techniques like XRD, EDS, FTIR, and SEM. X-Ray diffraction (XRD) spectra reveal the crystalline size of the sample. The qualitative and quantitative components of the ZnO-nanoparticles analyzed using Energy-dispersive X-Ray microscopy (EDS). The functional groups of the ZnO nanoparticles were investigated through Fourier Transform Infrared Spectroscopy (FTIR). The surface morphological structure of zinc oxide nanoparticles were obtained in Scanning Electron Microscopy (SEM). The Zinc oxide nanoparticles are mostly found and applied in field of medical science and anti-bacterial activities.

Keywords: Zinc Oxide nanoparticles; Elemental composition; Antibacterial.

1. INTRODUCTION

Nano means one-billionth. Thus nanotechnology deals with materials measured in a billionth of a meter. A nanometer is 1/80,000 the diameter of a human hair or approximately ten hydrogen atoms wide. Nanotechnology is the science of very small things. But nanotechnology is not just involved with small things. Nanotechnology is a multi-disciplinary science. It includes knowledge from biology, chemistry, physics, and other disciplines. Zinc oxide is an inorganic compound with the formula ZnO. It usually appears as a white powder, nearly insoluble in water. Zinc oxide is a multifunctional substance with its special physical and chemical properties, including high chemical stability, high electrochemical binding coefficient, wide spectrum of radiation absorption, and high photostability (Manjunatha et al. 2019).

Pesidium guajava (*P. guajava*) or the common guava is an evergreen shrub or small tree belonging to the family Myrtaceae. *P. guajava* is issued as a popular medicine against diarrhoea and is also used for wound dressing, ulcers, rheumatic pain. *P. guajava* leaves are also being reported to show antibacterial, anti-inflammatory, and anticancer properties.

P. guajava is selected for this study due to its high content of polyphenols (garlic acid, protocatechuic acid, caffeic acid, ferulic acid, chlorogenic acid, ellagic acid, guavin), flavanoids (quercetin, leucocyanidin,

kaempferol, quercetin-3-l-arabinofuranoside), carotenoids (carotene, lutein, lycopene, cryptoxanthin, rubixanthin, cryptoflavin, neochrome, phytofluene), triterpenes (oleanolic acid, ursolic acid, sitosterol, uvaol) present in the crude water extract of leaves. The aforementioned biomolecules have functional groups that can coordinate Zn (II) and will help in stabilization during the formation of ZnO nanoparticle (Saha et al. 2018).

2. MATERIALS & METHODOLOGY

Zinc oxide (ZnO) nanoparticles were prepared by using zinc acetate dehydrate ($C_4H_{10}O_6Zn$) and leaf extract of *Pesidium guajava* (Guava leaves) as a capping agent. The chemical reagents like NaOH solution, acetone, and deionized water were used.

2.1 Synthesis of ZnO Nanoparticles

10.975g of zinc acetate dehydrate was dissolved with 100ml of deionized water, and the solution was stirred for half an hour. The 10 ml of NaOH solution was added drop-wise to the above mixture to maintain the pH level 12. The final mixture was stirred for half an hour. The gelatinous precipitate was obtained. This obtained precipitate was aged for 24 hours and double washed with deionized water. Then the precipitate was kept in microwave oven for 30 minutes at 75 °C. And again furnace was also used to dry the material at 400 °C for four hours. The fine pure ZnO nanoparticles were collected by grained the dried cake using mortar.

2.2 Preparation of Plant Extract

Fresh and good quality leaves are collected, and it was washed in running tap water and then again washed using distilled water. These leaves were cut into small pieces using a knife. The 30g of chopped leaves were weighed and taken into a beaker with 100 ml of distilled water. Moreover, it was boiled for 30 minutes at 75 °C. By this time aqueous part turns green. The extract was filtered using Whatman No.1 filter paper to get a clear solution.

2.3 Synthesis of ZnO Nanoparticles using Capping agent (*Pesidium guajava*)

10.975g of zinc acetate dehydrate was dissolved with 100ml of deionized water and, the solution was stirred for half an hour for the reduction of zinc ions. 10ml of *Pesidium guajava* leaf extract was added into the 100ml of zinc acetate dehydrate solution. Then the mixture was stirred for half an hour using a magnetic stirrer. The 10 ml of NaOH solution was added drop-wise for the above mixture to maintain the pH level 12. The final mixture was stirred for half an hour. The gelatinous precipitate was obtained. This obtained precipitate was aged for 24 hours and double washed with deionized water. Then the precipitate was kept in the microwave oven for 30 minutes. The fine *Pesidium guajava* capped ZnO nanoparticles were collected by grained the dried cake using mortar.

3. CHARACTERIZATION TECHNIQUES

The prepared samples were characterized by using various techniques like Fourier transform infrared spectroscopy (FTIR), X-Ray diffraction (XRD), Scanning electron microscopy (SEM), Energy dispersive X-Ray spectroscopy (EDAX) and Anti-bacterial activity

3.1 FTIR

FTIR Spectroscopy is an analytical technique used to identify organic, polymeric, and, in some cases, inorganic materials. The FTIR analysis method uses infrared light to scan test samples and observe chemical properties.

3.2 XRD

X-ray diffraction (XRD) is a technique used in materials science for determining the atomic and molecular structure of a material. This is done by irradiating a sample of the material with incident X-rays and then measuring the intensities and scattering angles of the X-rays that are scattered by the material.

3.2.1 Grain Size

The crystalline size calculated by using the Debye scherrer formula is given by,

$$D = k\lambda/\beta \cos \theta$$

Where, k - Wavelength of XRD, λ - Full width half maximum, θ - Bragg's angle, k - Constant

3.2.2 Lattice constants and unit cell volume:

The lattice parameter calculated by the equation of

$$1/d^2 = (4(h^2+hk+k^2)/3a^2) + (l^2/c^2)$$

Where, d - Plane spacing a , c - the lattice parameters which confirm the hexagonal structure.

The unit cell volume (V) of the sample is given by the below equation:

$$V = (\sqrt{3}/2) \times a^2 \times c \quad (5.3)$$

3.3 SEM

SEM analysis is a powerful investigative tool that uses a focused beam of electrons to produce complex, high magnification images of a sample's surface topography.

3.4 EDAX

Energy Dispersive X-Ray Analysis (EDX), is an x-ray technique used to identify the elemental composition of materials. Applications include materials and product research, troubleshooting, deformation, Environmental pollutant analysis and more.

4. RESULTS

4.1 FTIR Analysis

Fourier Transform Infrared Spectroscopy (FTIR) identifies chemical bonds in a molecule by producing an infrared absorption spectrum. The FTIR spectrum of the prepared pure and capped ZnO samples were plotted between the wavelength ranges about 4000-400 cm^{-1} is shown in fig 1. The verity of peaks is absorbed in the different wavenumbers. A large number of absorption peaks is 3848.11 cm^{-1} and 3854.96 cm^{-1} it corresponds to the O=H stretching banded (alcohol). The peak at 3441.73 cm^{-1} and 3637.40 cm^{-1} indicates the N=H bond (Amine). The absorbance at 2800 cm^{-1} and 2793.15 cm^{-1} it has represented the C=H bond (Alkynes). The absorption peaks at 1657.46 cm^{-1} and 1677 cm^{-1} it indicates the presence of C=HO stretching (Hydrogen). And then N=O stretching (Nitro) absorption represents the peak at 1413.91 cm^{-1} and 1441.27 cm^{-1} , respectively. Those results are indicating that the

synthesized ZnO nanoparticles are stabilized by biomolecular constituents present in the leaf extract of *Pesidium guajava* is shown in Table 1.

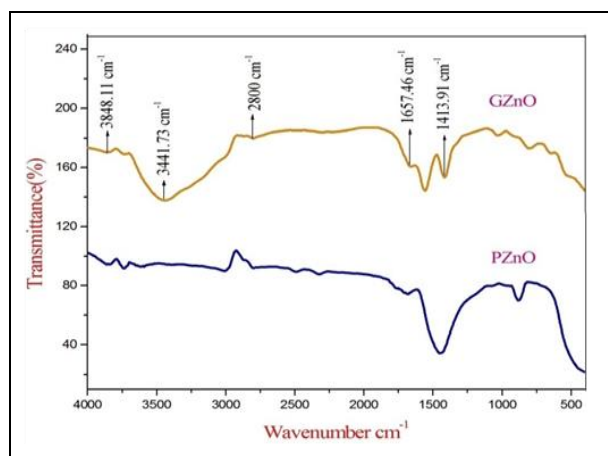


Fig. 1: FTIR spectrum of Pure and capped ZnO nanoparticles

4.2 XRD Analysis

XRD is primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized, and the average bulk composition is determined. The XRD pattern of prepared PZnO and GZnO were shown in fig 2. The prepared sample confirms the presence of hexagonal structure. The diffraction peaks of the prepared PZnO at $2\theta=36.44$, 63.03 , 68.1 , and 69.29 then the diffraction peaks of GZnO at $2\theta=36.647$, 63.056 , 67.192 and 69.222 are identified. Both are corresponding to the hkl planes are (101), (103), (112), and (201). The average crystalline size (D) of PZnO and GZnO was 18.67 and 14.26 nm. Thus the average crystalline size of PZnO is higher than the GZnO due to the capping of leaf extract. The unit cell volume (V), lattice parameters a and c decreased due to an increase in crystalline size, and it is shown in table 2.

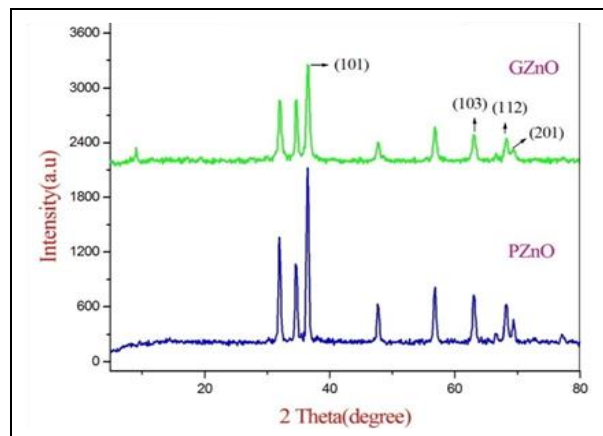


Fig. 2: XRD Pattern of Pure and Capped ZnO Nanoparticles

4.3 SEM Analysis

The morphological analyses of the synthesized materials are analyzed by scanning electron microscope (SEM). The two-dimensional SEM images of pure and capped zinc oxide nanoparticles are shown in fig 3. The pure ZnO nanoparticles (a) were given the needle structure. Moreover, a capped ZnO nanoparticle (b) shows the spherical structure. The particle size of pure ZnO was 25.45 to 103.36 nm, and capped ZnO nanoparticles are 47.27 to 76.45 nm.

4.4 EDAX Analysis

Energy-dispersive X-ray spectroscopy (EDX) provides a qualitative and quantitative analysis of the prepared sample. Moreover, it's also used to calculating the chemical composition of the elements. The functional groups of Zn and O are present in the sample. The atomic weight for Zn in PZnO was 40.54% , and O was 59.46% . And the atomic weight of Zn in GZnO was 35.51 and O is 64.49 , respectively. The EDX analysis of PZnO and GZnO is shown in fig 4.

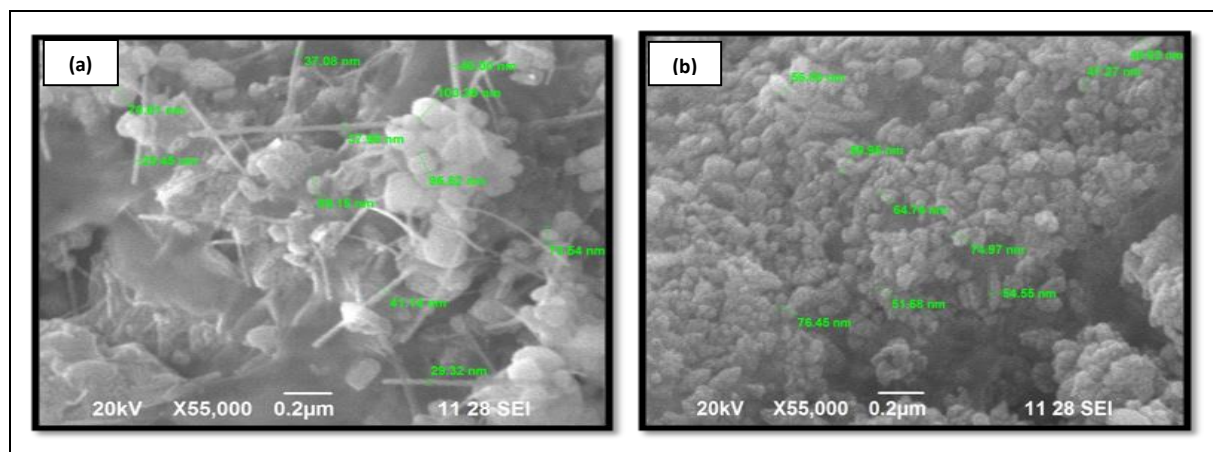


Fig. 3: SEM Analysis for Pure and Capped ZnO (a) Needle Structure (Pure ZnO) (b) Spherical Structure (Capped ZnO)

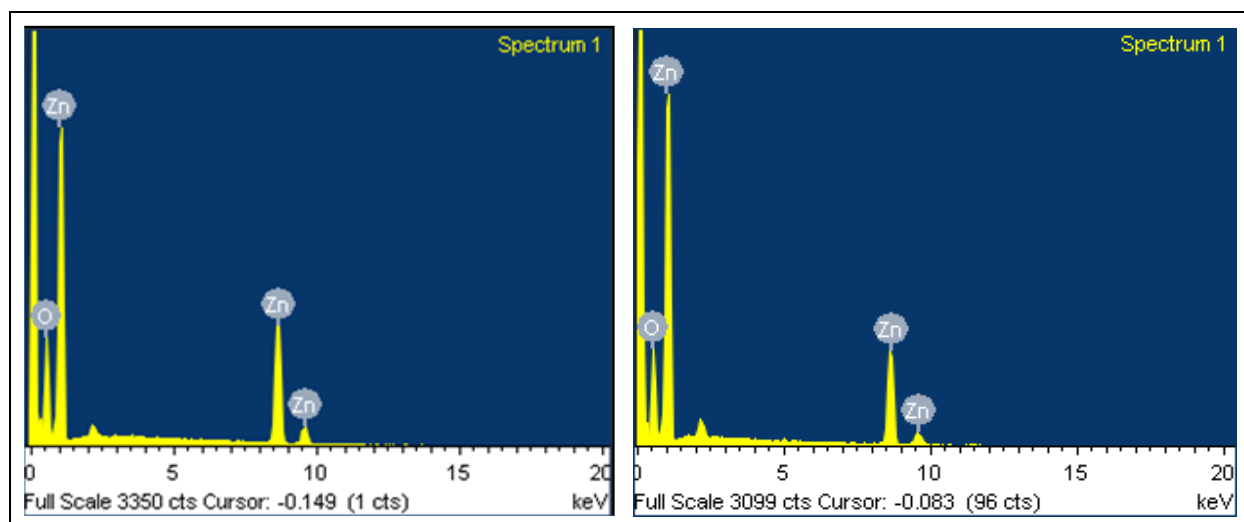


Fig. 4: EDAX Analysis for Pure and Capped ZnO

6. CONCLUSION

In this research work, the zinc oxide nanoparticles were synthesized using with and without capping agent *Pesidium guajava* (guava) leaf extract. The samples were analyzed by FTIR, XRD, SEM, and EDX.

- The FTIR analyses reveal the different functional groups are present in the given pure ZnO and capped ZnO.
- XRD pattern shows the presence of hexagonal structure, and it is used to determine the crystalline structure of the sample and unit cell dimensions of the prepared sample PZnO and GZnO.
- SEM confirms the morphological structure of the prepared sample. Moreover, it gives the Needle shapes for the pure ZnO and spherical structure for the Capped ZnO, so the SEM analysis reveals that the capping agent can change some morphological structure of the given samples.
- EDX confirms that the foreign elements are present in the prepared samples. The PZnO and GZnO nanoparticles are having zinc (Zn) and oxide (O)

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